CONFIDENTIAL

Copy
RM E57B15

C. 1

NACA RM E57B15

NACA

RESEARCH MEMORANDUM

BONDED LEAD MONOXIDE FILMS AS SOLID LUBRICANTS FOR TEMPERATURES UP TO 1250° F

By Harold E. Sliney and Robert L. Johnson

Lewis Flight Propulsion Laboratory
Cleveland, Ohio

CLASSIFICATION CHANGED
UNCLASSIFIED

To ________________

NACA Res abs effective

By authority of VRN-127 Date May 16, 1958
AMT 7-4-58

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS
WASHINGTON
May 7, 1957

CONFIDENTIAL

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

RESEARCH MEMORANDUM

BONDED LEAD MONOXIDE FILMS AS SOLID LUBRICANTS FOR TEMPERATURES UP TO 1250° F

By Harold E. Sliney and Robert L. Johnson

SUMMARY

A friction, wear, and endurance-life study was made with bonded films of mixed oxides containing lead monoxide (PbO) as the main component. The solid-lubricant films were bonded to low-carbon-steel and stainless-steel disks. Hemispherical tipped (3/16-in. rad) M-1 tool-steel riders were run against the coated flat surfaces of disks at a sliding velocity of 430 feet per minute at temperatures up to 1250° F. Cast Inconel riders were also used for runs above 1000° F.

The coatings lubricated over the entire temperature range, but were far more effective from 500° to 1250° F than at the lower temperatures. Improved lubrication at the higher temperatures was apparently associated with the formation of a glazed wear track on the coated disks.

The endurance life of the coatings in high-load friction tests was much better from 500° to 1250° F than at lower temperatures. However, the endurance life at room temperature was at least as good as that of a resin-bonded molybdenum disulfide (MoS_2) lubricant film. Friction was somewhat higher at all temperatures than for MoS_2 at room temperature.

Bonded coatings with PbO as the primary component appear promising for use as high-temperature solid-lubricant films. In addition to good lubricating properties at elevated temperatures, these bonded coatings appear to have sufficiently good room-temperature properties for use in applications requiring cycling through a wide temperature range.

INTRODUCTION

A real need exists in aircraft applications for lubricants to function in oxidizing atmospheres at temperatures of 400° to 1000° F and higher. Aerodynamic heating, as well as heat from combustion in engines, can produce these high-temperature levels. Problem areas that are of

[redacted]

particular significance include the bearings for thrust-reversal devices in jet transports, various operating parts including bearings in jet engines, and pivot bearings for primary control surfaces on supersonic aircraft and missiles.

Solid lubricants are perhaps the most promising type of lubricant for use at extreme temperature conditions. The most effective way of using solid lubricants has been by forming bonded surface films. Previous research by NACA (ref. 1) showed yellow lead monoxide (PbO) powder to be an effective solid lubricant at 1000° F. At lower temperatures (700° to 900° F), however, PbO was converted by oxidation into red lead oxide (Pb_3O_4). The maximum rate of this conversion process occurs at 800° F (ref. 2). Contamination of PbO by Pb_3O_4 was harmful to its lubricating effectiveness in the experiments of reference 1. Fortunately, PbO is the most stable high-temperature (>950° F) form of lead oxide, and at temperatures above 1000° F the contaminating Pb_3O_4 will revert to PbO. 4404

The high-temperature reversion of Pb_3O_4 to PbO reduces the severity of the problem of Pb_3O_4 contamination. The presence of Pb_3O_4 in PbO at any time, however, is undesirable because it reduces lubricating effectiveness. Several methods have been considered for inhibiting the oxidation of PbO to Pb_3O_4. One method of inhibiting oxidation is to disperse PbO particles throughout a second phase that will physically shield them from oxidizing atmospheres.

The research reported herein was performed to formulate, apply, and evaluate ceramic-type solid-lubricant coatings for metal surfaces consisting primarily of PbO dispersed in a matrix believed to be tetralead silicate. The coatings were intended to be capable of preventing surface failure, excessive wear, and high friction between sliding surfaces. Several experimental PbO coatings were prepared using low percentages (5 to 10 percent) of silicon dioxide (SiO_2) to form the matrix. The properties of the coatings were studied by mechanical, metallographic, and chemical analyses, and X-ray diffraction techniques. Friction, wear, and endurance data were obtained at temperatures up to 1250° F.

MATERIALS

Raw Materials for Coatings

The coatings evaluated in these experiments were prepared from mixed powders of PbO and SiO_2. Both oxides were certified reagent grades with particle sizes of about 200 mesh.

Test Specimens

The rider specimens for all tests run at temperatures up to 1000° F were M-1 tool steel hardened to Rockwell C-60. Both M-1 and cast Inconel riders were used at higher temperatures. The disk specimens (2.5-in. diam, 0.5-in. thickness) to which the coatings were fused were SAE 1020 steel and 440-C stainless steel. The rider specimens were cylindrical (3/8-in. diam and 3/4-in, length) and had a hemispherical tip (3/16-in. rad) ground on one end. Before the coatings were applied, the surfaces of the disks were ground flat and had a roughness of 4 to 8 rms.

APPARATUS

A diagrammatic sketch of the apparatus used to determine friction, wear, and endurance properties of the solid-lubricant coatings is shown in figure 1. The basic elements of this apparatus were the rotating disk specimen and the cylindrical rider specimen with a hemispherical contact tip. The rotating specimen was driven through a belt system by an electric motor coupled with a variable-speed power transmission device. This arrangement allows good speed control with a range of sliding velocities up to approximately 500 feet per minute. Loading was obtained by the use of dead weights to apply force through the pulley system shown in figure 1. The net loads on the sliding contact were from 200 to 2000 grams (with steel specimens, these loads given initial Hertz surface stresses from 87,000 to 188,000 psi). The disk and rider specimens were operated in an Inconel pot. The pot has strip heaters mounted on its periphery and ring heaters under its base, and thus served as a furnace. Temperatures up to 1250° F can be obtained in the furnace pot.

The rider-holder assembly was located by rolling contact bearings with the shaft free to move axially and to rotate within the bearing housing. The apparatus was insensitive to the very small friction losses in the bearings. Contact of the rider specimen with the disk provided axial location of the rider-holder assembly when the load was applied. Friction force was measured by four strain gages mounted on a copper-beryllium dynamometer ring. The strain gages were part of a Wheatstone bridge circuit. The friction coefficients reported are the ratios of friction force to applied normal load. Friction coefficients were usually reproducible within ±0.02.

PROCEDURE

Coating

The following procedure was used in coating the disks and preparing them for testing:

(1) Cleaning prior to coating
  (a) Wash steel disk with acetone.
  (b) Scrub with repeated applications of moist, levigated alumina.
  (c) Wash in tap water.
  (d) Wash in distilled water.
  (e) Blot dry with bibulous paper.

(2) Preoxidation
  (a) Heat disk in air at 1650° F for 3 to 5 minutes, or until a thin, uniform, yellow or pale blue oxide film is apparent. 4404
  (b) Cool in air to room temperature.

(3) Powder application
  (a) Individual batches of powders containing 5 percent, 7.5 percent, and 10 percent silica, with the balance in each case of yellow PbO, were prepared by weighing the proper proportions for each on an analytical balance. Mixing bottles were one-third filled with the weighed portions and mixed by vigorously shaking the bottles for at least 15 minutes.
  (b) A thin, uniform layer of the mixed oxide powder was applied to the surface of the preoxidized disk by brushing the dry powder through a No. 40 U.S. Standard sieve held about 10 inches over the disk. This method resulted in much more uniform coatings than previous attempts to apply the powder as acetone or water slurries.

(4) Firing
  (a) The disks were placed in a 1650° F furnace for 6 to 7 minutes or until the mixed powder melted and formed a uniform molten film. The disks had to be perfectly level in order to obtain a reasonably uniform melt. The viscosity and surface tension of the molten oxides were sufficiently high to retain melts up to 0.020 inch thick on the surface of a flat disk.
  (b) The disks were then removed from the furnace and placed on a level steel block and allowed to cool in room air.

(5) Finishing: The coatings were finish ground to the desired thickness.

Cleaning

Prior to each run, both the coated disks and the riders were cleaned according to the following procedure:

(1) Wash with acetone.

(2) Scrub with repeated applications of moist, levigated alumina. This step was omitted in preparing the disks to avoid embedding alumina in the coatings.

(3) Wash in tap water.

(4) Wash in distilled water.

(5) Wash in 95-percent ethyl alcohol.

(6) Dry in warm airstream and store in a desiccator.

4404

Testing

The coatings on all the disks for which friction, wear, or endurance life data are reported were about 0.0040 inch thick with the exception of one series of coatings bonded to 440-C stainless-steel disks. These coatings were finish ground to various thicknesses from 0.0005 to 0.0065 inch.

All wear tests were run in dry air with the slider specimen under a 1-kilogram load and at a sliding velocity of 430 feet per minute. All endurance tests were run under the same conditions, but a 2-kilogram load was employed. During the first 10 minutes of the endurance tests, the specimens were "run-in" at gradually increased loads. The sequence was as follows: 2 minutes at 200 grams, 2 minutes at 400 grams, 2 minutes at 600 grams, 2 minutes at 1000 grams, 2 minutes at 1600 grams, and finally 2000 grams for the balance of the test. The temperature was held constant, and the friction was recorded continuously during each test. The specimens were held at test temperature for at least 1/2 hour before each run.

RESULTS AND DISCUSSION

In this work, methods were developed for bonding PbO-base ceramic coatings to low-carbon steel and stainless-steel surfaces by means of a fusion process similar to that employed in applying porcelain enamels to metal surfaces (ref. 3). Other compounds present in the coatings were silica (SiO_2), as an additive constituent, and magnetite (Fe_3O_4) formed by oxidation of the base metal. The compositions of the PbO-base coatings will be discussed in detail later.

Effect of Silica Additions on Coating Formation

Small percentages of silica were incorporated into the composition of PbO coatings. Silica was selected for the formulations because it has desirable phase relations (eutectics) with PbO and also promotes vitrification. Therefore, silica additions would both lower the melting point and broaden the temperature range for softening. Lower melting point and lower hardness are associated with reduced shear strength. It was considered, therefore, that silica additions could contribute to low friction.

Figure 2 shows that pure PbO melts at 1628° F; however, an eutectic is formed with 8.22 percent by weight of silica and PbO. This eutectic melts at 1317° F and has a lower temperature range for softening than pure PbO. Greater plasticity within the temperature range for softening increases the probability of self-repair in damaged films and thereby 4404
can improve endurance life.

When melts containing less than 6.7 weight percent silica are cooled, the first phase to solidify is PbO. After complete solidification, discrete particles of PbO are dispersed throughout a tetralead silicate ($4PbO\text{-}SiO_2$) phase (refs. 4 and 5). Several advantages may thereby be derived, for example,

(1) The silicate phase functions as a binder, and increases the strength of the coating

(2) The silicate phase surrounding the PbO particles protects them from oxidizing atmospheres, thus minimizing the conversion to higher oxides

(3) The presence of silica increases the range of softening temperatures and the vitrification or the "glass-forming" tendency of the coating.

A pronounced vitrification tendency promotes the formation of glazes on the ceramic wear track. It has been observed that the friction and wear of metals in sliding contact with ceramics generally decrease sharply when a glazed wear track forms on the ceramic surface (refs. 6 and 7). It is also interesting to note that molten glass has been successfully used as a die lubricant in the extrusion of metals (ref. 8).

The phase diagram for the $PbO\text{-}SiO_2$ system (fig. 2) shows that free PbO does not exist in fused mixtures containing more than 6.7 percent by weight of SiO_2, but is combined with silica in the form of tetralead silicate, orthosilicate, etc. (ref. 4). However, such a combination is only strictly true for melts cooled very slowly throughout the liquidus temperatures. By rapidly quenching the melts, it was possible to disperse free PbO in coatings prepared by the fusion of mixed powders containing up to 10 percent SiO_2. In addition, the system was complicated by the presence of oxides of the coated metal, primarily Fe_3O_4, which formed during the firing process and diffused into the melt. Coatings containing more than 10 percent silica had poor thermal shock resistance, and were considered too brittle for bearing applications. Coatings containing 5 to 10 percent silica adhere to the metal surfaces when thermally cycled in air between 70° and 1500° F.

Determination of Coating Composition

X-ray diffraction studies were conducted to determine the compounds
4404 present in the fused coatings. All quantitative determinations were obtained by chemical analyses.

The PbO form of lead oxide was apparently stabilized in the coatings. X-ray analyses invariably showed a strong α-PbO pattern. No distinct pattern for any other form of lead oxide was ever found, even in coatings that had been previously held for a considerable time in the temperature range at which rapid oxidation to Pb_3O_4 normally would be expected. X-ray patterns further showed that iron oxide was present in the coatings, primarily as Fe_3O_4.

Coatings applied to SAE 1020 steel contained 20 to 30 percent Fe_3O_4; those applied to 440-C stainless steel contained 5 to 10 percent Fe_3O_4 and 0.1 to 0.8 percent chromium, probably present as an oxide. (Results of X-ray and chemical analyses are summarized in table I.)

Effect of Coating Thickness on Friction and Wear

The coatings were generally about 0.0150 inch thick after firing. They were finish ground to the desired thicknesses and to a surface finish of about 20 rms. All thicknesses reported herein are considered nominal except for those given in table II. All others were obtained by subtracting the thickness of the uncoated disk from that of the finished, coated disk. During the fusion process, the molten oxides may etch the base metal to a depth of several ten-thousandths of an inch. Consequently, the actual coatings are probably slightly thicker than the nominal values given.

Preliminary friction and wear tests indicated that the thickness of coatings applied to SAE 1020 steel should be greater than 0.0010 and less than 0.0070 inch (table II). Coatings thinner than 0.0020 inch failed early because of penetration by the rider specimen. Early failure also occurred with coatings thicker than 0.0070 inch. Photomicrographs (figs. 3(a) and (b)) show that the coatings generally consisted of two distinct layers. The transition layer immediately adjacent to the base metal is of irregular thickness, averaging about 0.0010 inch, and is high in iron oxide content. This intermediate layer is apparently of little value as a solid-lubricant film. Therefore, grinding the coatings down to a thickness approaching 0.0010 inch essentially removes the solid lubricant and exposes a film having poorer lubricating characteristics. Figure 3(b) is a cross section through the wear track of a coating originally 0.0030 inch thick, and shows that, even after prolonged testing, the rider had not penetrated to the intermediate layer. Figure 3(c) shows the surface of the coating and the wear track after a test.

Figure 4(a) is a cross section of a bonded PbO coating on 440-C 4404
stainless steel. Figure 4(b) is a cross section of a wear track on the same coating. It can be seen that the intermediate layer is less than one-thousandth of an inch thick. Some coatings were ground to a nominal thickness of 0.0005 inch, and they still maintained a lubricating film. Figure 5 illustrates the effect of coating thickness on the friction and wear of cast Inconel sliding against coated 440-C disks in a 1250° F air atmosphere. The rider wear rate was low for all thicknesses between 0.0005 and 0.0040 inch, but increased sharply for thicker coatings. Friction was 0.10 for the thinnest coating, increased to 0.25 for the 0.0040-inch coating and to 0.30 for the 0.0065-inch coating. Figure 6 consists of photomicrographs of the wear tracks produced on coatings of various thicknesses and of the wear areas produced on the corresponding cast Inconel riders.

Apparently, the coatings should be as thin as possible consistent with satisfactory endurance life and complete coverage of the intermediate layer with a solid-lubricant film. This observation agrees with the theory of Bowden and Tabor that the frictional force consists essentially of a shearing term and a ploughing term (ref. 9). During sliding, friction can be attributed to the force required to either shear a lubricating film interposed between the sliding surfaces, or in the absence of a lubricant, to the force required to shear metallic junctions formed by welding of surface asperities. In addition, if one material is harder than the other, the harder surface asperities penetrate and plough through the softer material, thus adding to the total frictional force, the force required to displace the softer material. Therefore, keeping the coating thickness to a minimum and utilizing a relatively hard substrate minimizes the ploughing term and reduces friction.

Although the intermediate layer is not in itself a good solid lubricant, its presence is probably required to obtain a good bond between the base metal and the coating. This intermediate layer is essentially a thin layer of scale, consisting principally of oxides of the base metal. In reference 10 it is shown that during the firing of enamels on steels, a thin "hammer-scale" of Fe_3O_4 forms, which grows fast to the steel surface and is intermeshed in the enamel. It was further demonstrated that the coefficient of expansion of this scale is intermediate between that of steel and most enamels (ref. 11). Therefore, the intermediate layer is a transition layer which imparts good thermal shock resistance to the coatings. However, NACA experience indicates that the scale layer must not be too thick, or it will tend to spall away from the base metal when the coating is stressed by grinding or by the pressure of the slider specimen. For a given base metal, the thickness of the transition layer and the amount of iron oxide which diffuses into the surface layer can be controlled to some extent by the time and temperature at which the base metal is preoxidized and by the time and temperature of the firing operation. Iron oxide imparts hardness and strength to the coatings, but it appears that the amount must be kept below about 30 percent to avoid excessive brittleness. These considerations demonstrate that time and temperature employed in the coating procedure must be rather closely controlled.

Effect of Temperature on Friction and Wear

Friction and wear tests were run at various temperatures, using a 1-kilogram normal load, and a sliding velocity of 430 feet per minute. The PbO coating thicknesses were 0.0040 and 0.0010 inch. Figure 7 presents wear and friction data for the coatings. Both wear and friction approached a minimum at 500° F, and wear increased somewhat at higher temperatures. At 1250° F, the average friction coefficient for M-1 in sliding contact with the thicker coating was 0.31 and for cast Inconel, 0.25; the thin coating gave a friction coefficient for cast Inconel of 0.14. The curves of figure 7 indicate that for temperatures of 500° F and higher the thin coating gave a little over one-half the friction obtained with the thicker coating. The friction of M-1 tool steel in sliding contact with SAE 1020 steel disks did not vary significantly with the iron oxide content and only slightly with the silica content within the composition limits given in table I.

Reference data (fig. 7) were obtained using disk specimens coated with a typical commercial-type resin-bonded MoS_2 film. The MoS_2 coatings were believed to be about 0.0003 inch thick. The MoS_2 film gave lower wear and friction than the PbO films at temperatures below 500°. At temperatures above 600° F, however, friction and wear with the MoS_2 film increased significantly and the PbO film provided more effective lubrication. It should be noted that 600° F is the maximum temperature at which the use of this particular MoS_2 film is recommended by the supplier.

Perhaps the most interesting feature of the bonded films was their ability to prevent galling and excessive wear of the metal surfaces over a wide temperature range (70° to 1250° F). Their effectiveness in this regard was most pronounced between 500° and 1000° F. Figure 7 shows the wear rate of the riders at various temperatures. Wear was quite low at room temperature and 1250° F and was negligible between 500° and 1000° F. A glaze formed on both the riders and wear track between 500° and 1250° F. The coatings were depressed one- or two-thousandths of an inch in the wear track, but figures 3(b) and 4(b) illustrate that neither metallic contact, nor penetration to the intermediate layer had occurred at the conclusion of the 1-hour tests.

At 1250° F, the wear rate of cast Inconel bullets was the same as that of M-1 tool steel. For comparison, the effect of temperature on coefficient of friction of phenol-impregnated carbon against cast iron (ref. 12) are included in figure 7. In interpreting friction data, the trends obtained are more important than the absolute values. Isolated data points may be misleading, particularly in the endurance runs.

4404

Endurance Properties

Endurance runs were made by continuously sliding a hemispherically tipped rider under a normal load of 2 kilograms over the same wear track at a rate of 825 cycles per minute. Endurance life was reported as the number of cycles the rider specimen passed over the same wear track before failure. Failure was taken as the time at which the friction rose sharply and remained at a high value. The tests were stopped at 240,000 cycles if failure did not occur up to that point.

Tables III and IV give the endurance life of the 0.0040-inch coatings at various temperatures. For comparison, at room temperature the number of cycles before failure was about twice that previously reported for the best resin-bonded MoS_2 films using identical methods of evaluation at the Lewis laboratory (ref. 13). Between 500° and 1250° F, the number of cycles to failure increased at least four times beyond the room-temperature life. Thus, at the higher temperatures, the endurance lives of the PbO coatings were at least eight times those previously reported for MoS_2 coatings at room temperature.

Table III also shows that, at temperatures below 1000° F, the endurance life of the coatings decreased with increasing silica content. This decrease can be attributed to the embrittling effect of the silica. The maximum endurance was obtained with coatings prepared from powders containing 5 percent SiO_2. The use of thinner films may further improve the endurance life of the coatings.

Table V gives scratch hardness values for 0.004-inch coatings of various compositions.

SUMMARY OF RESULTS

In an investigation of methods of forming PbO-base solid-lubricant coatings, and in evaluating the friction, wear, and endurance properties of the coatings, the following results were obtained:

1. A procedure was developed for coating low-carbon steel and stainless steel with bonded films of high lead monoxide (PbO) content. These films functioned as effective solid lubricants from 70° to 1250° F, but were especially effective from 500° to 1250° F. A glazed wear track invariably formed on the coatings tested at temperatures of 500° F and higher. The coatings were usually about 0.0040 inch thick although this is not considered the optimum film thickness for all metals.

2. The wear rate of M-1 tool steel riders in sliding contact with coated 440-C stainless steel and SAE 1020 steel disks was moderate at room temperature, but extremely low from 500° to 1000° F. For a given coating thickness, friction decreased as the temperature was increased from 70° to 500° F. Friction was practically constant from 500° to 1000° F. Cast Inconel riders and coated 440-C stainless-steel disks were used at 1250° F. Rider wear was moderate, and the average coefficient of friction was 0.25 for a 0.0040-inch coating and 0.14 for a 0.0010-inch coating.

3. At 1250° F, friction was significantly lowered (from 0.25 to 0.10), and rider wear was significantly reduced by decreasing film thickness from 0.0040 to 0.0005 inch.

4. The endurance life of 0.0040-inch thick coating under high load was at least four times greater at temperatures above 500° F than at room temperature. Also, the room-temperature life was at least as good as that of bonded MoS_2 films tested under identical conditions.

5. The coatings were applied by melting mixtures of silica (SiO_2) and PbO directly on the base metal. Small percentages of SiO_2 were included in the composition of the coatings to increase their strength, hardness, and glaze-forming tendencies, and to serve as an oxidation barrier for the PbO. Iron oxide was also present in the coatings. It formed by oxidation of the base metal during the fusion process, and diffused into the PbO-SiO_2 melt. Iron oxide increased the hardness of the coatings and promoted good bonding to the base metal.

The coatings may be classified as ceramics, because they were formed by the fusion of mixed oxides. They adhered well to the base metal and were capable of withstanding considerable thermal shock without spalling. Hardness depended upon the silica and iron oxide content. The scratch hardnesses of the coatings were higher than those of mild steel but much lower than those of hardened tool steel.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, February 19, 1957

REFERENCES 4404

1. Peterson, M. B., and Johnson, R. L.: PbO and Other Metal Oxides as Solid Lubricants for Temperatures to 1000° F. Preprint No. 56LC-10, ASLE, 1956.

2. Mellor, J. W.: A Comprehensive Treatise on Inorganic and Theoretical Chemistry. Vol. VIII. Longmans, Green and Co., 1940, p. 673.

3. Andrews, A. I.: Enamels. The Twin City Printing Co. (Champaign, Ill.), 1935.

4. Levin, Ernest M., McMurdie, Howard F., and Hall, F. P.: Phase Diagrams for Ceramists. The Am. Ceramic Soc., Inc., 1956, p. 59.

5. Geller, R. F., Creamer, A. S., and Bunting, E. N.: The System: PbO-SiO_2. Jour. Res. Nat. Bur. Standards, vol. 13, no. 2, Aug. 1934, pp. 237-244.

6. Coes, L., Jr.: Chemistry of Abrasive Action. Ind. and Eng. Chem., vol. 47, no. 12, Dec. 1955, pp. 2493-2494.

7. Herron, R. H.: Friction Materials - A New Field for Ceramics and Cermets. Am. Ceramic Soc. Bull., vol. 34, no. 12, Dec. 1955, pp. 395-398.

8. Sejournet, J., and Delcroix, J.: Glass Lubricant in the Extrusion of Steel. Lubrication Eng., vol. 11, no. 6, Nov.-Dec. 1955, pp. 389-396.

9. Bowden, F. P., and Tabor, D.: The Friction and Lubrication of Solids. Clarendon Press (Oxford), 1950, p. 90.

10. Dietzel, A., and Meures, K.: Reactions Important for Adherence When Firing Ground Coats Containing No Adherence-Promoting Oxide. Jour. Am. Ceramic Soc., vol. 18, no. 2, Feb. 1935, pp. 35-36.

11. Dietzel, A., and Meures, K.: The Adjustment of Enamels to Sheet Steel. Jour. Am. Ceramic Soc., vol. 18, no. 2, Feb. 1935, pp. 37-38.

12. Johnson, Robert J., Swikert, Max A., and Bailey, John M.: Wear of Typical Carbon-Base Seal Materials at Temperatures to 700° F. NACA TN 3595, 1956.

13. Godfrey, Douglas, and Bisson, Edmond E.: Bonding of Molybdenum Disulfide to Various Materials to Form a Solid Lubricating Film. II - Friction and Endurance Characteristics of Films Bonded by Practical Methods. NACA TN 2802, 1952.

4404

TABLE I. - RESULTS OF X-RAY DIFFRACTION STUDIES AND

CHEMICAL ANALYSES OF PbO-BASED COATINGS

Powder composition prior to firing, percent by weight		Base metal	Typical coating compositions after firing at 1650° F on base metal, percent by weight				
SiO_2	PbO		PbO	Fe_3O_4	SiO_2 (a)	Cr_2O_3	Unaccounted for
5	95	SAE 1020 steel	62.7	29.0	3.1	---	5.2
		440-C stainless steel	86.0	5.9	4.9	0.2	3.0
7.5	92.5	440-C stainless steel	71.5	10.5	7.0	1.5	9.5
10	90	SAE 1020 steel	69.5	24.3	8.1	---	High by 1.9

[a]SiO_2 determined only by chemical analyses. SiO_2 probably combined with PbO in coatings as $4PbO\text{-}SiO_2$.

4404

TABLE II. - LIFE OF 95 PERCENT PbO AND 5 PERCENT

SiO_2 COATINGS ON SAE 1020 STEEL AS A

FUNCTION OF COATING THICKNESS

[Thickness measured with micrometer depth gauge (metal surface to coating surface at a failure area).]

Thickness mils, (thousandths of in.)	Qualitative rating of life
16	Very poor
11	Very poor
9	Very poor
9	Poor
7	Poor
4	Good
4	Good
4	Good
1	Poor

[redacted]

TABLE III. - RESULTS OF ENDURANCE TESTS OF 0.0040-INCH-THICK $PbO-SiO_2$ COATINGS

[2-Kg load; 430 ft/min; M-1 riders.]

4404

Temperature, °F	Coating materials (composition before firing), percent by weight					
	5 SiO_2; 95 PbO		7.5 SiO_2; 92.5 PbO		10 SiO_2; 90 PbO	
	Endurance life, cycles before failure	Coefficient of friction at 1 hr (49,500 cycles)	Endurance life, cycles before failure	Coefficient of friction at 1 hr	Endurance life, cycles before failure	Coefficient of friction at 1 hr
70	50×10^3	0.42			30×10^3	[a]0.30
250	50	.25				
500	>240	.26	110×10^3	0.24	90	.20
750	>240	.33				
1000	>240	.30	>240	.14	>240	.25
1250	>240	[b].26				

[a]Coefficient of friction at 30 min.

[b]Cast Inconel rider; disk base metal, 440-C stainless steel.

TABLE IV. - MISCELLANEOUS RESULTS OF ENDURANCE TESTS

Rider	Disk	Coating	Cycles to failure	Coefficient of friction at 1 hr	Temperature, °F
[a]SAE 1095	SAE 1020	0.0002 to 0.0005 In. thick resin-bonded MoS_2 film	20×10^3	----	75
M-1	SAE 1020	Uncoated	-------	0.50	500
M-1	SAE 1020	$10BN-10SiO_2-80PbO$	180	.32	500

[a]Data for SAE 1095 rider against SAE 1020 disk coated with MoS_2 from ref. 13.

4404

TABLE V. - SCRATCH HARDNESS VALUES OF TYPICAL PbO-BASE COATINGS COMPARED WITH STANDARD METAL TEST BLOCKS

Powder composition prior to firing, percent by weight	Base metal	Surface finish, rms micron (a)	Scratch hardness[b]	
			Scratch width in microns, λ	Bierbaum micro-character, K
Uncoated	Standard steel test block, hardness, Rockwell C-64	12-15	4.0	2500
Uncoated	SAE 1020, hardness, Rockwell B-85	10-12	10.0	400
5 SiO_2-95 PbO	SAE 1020	(20)	8.4	568
7.5 SiO_2-92.5 PbO	SAE 1020	(20)	7.0	820
10 SiO_2-90 PbO	SAE 1020	(10)	5.8	1190
5 SiO_2-95 PbO	440-C Stainless steel	(20)	7.8	657

[a]Values of rms in parentheses are estimated values; soft, microscopic pockets of PbO were penetrated by profilometer scribe to give unreasonably high rms values.

[b]$K = 10^4/(\lambda/2)^2$ = microcharacter. (Scratch made by 101° diamond point under a normal load of 9 g.)

4404

CA-3

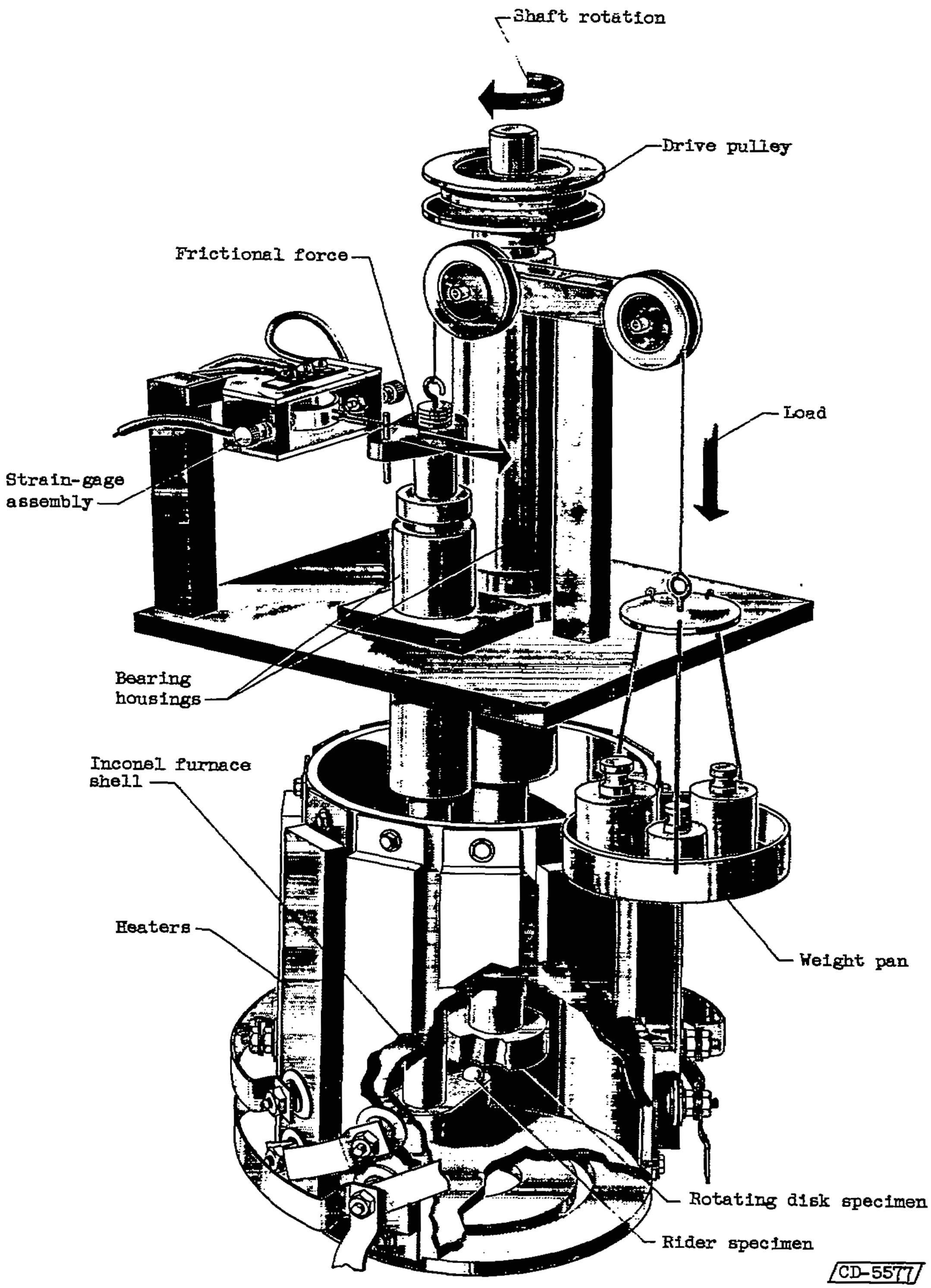


Figure 1. - Friction apparatus.

4404

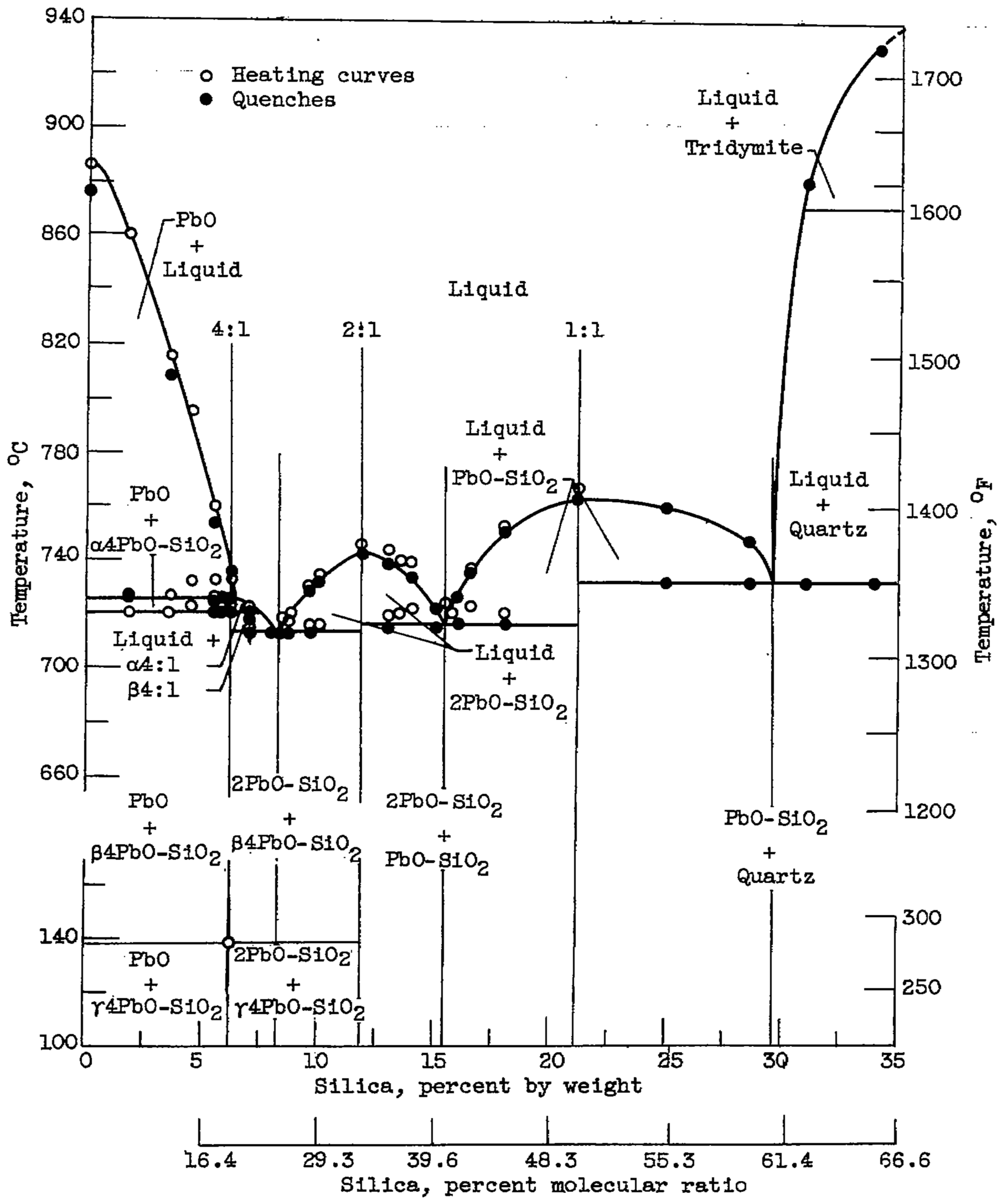
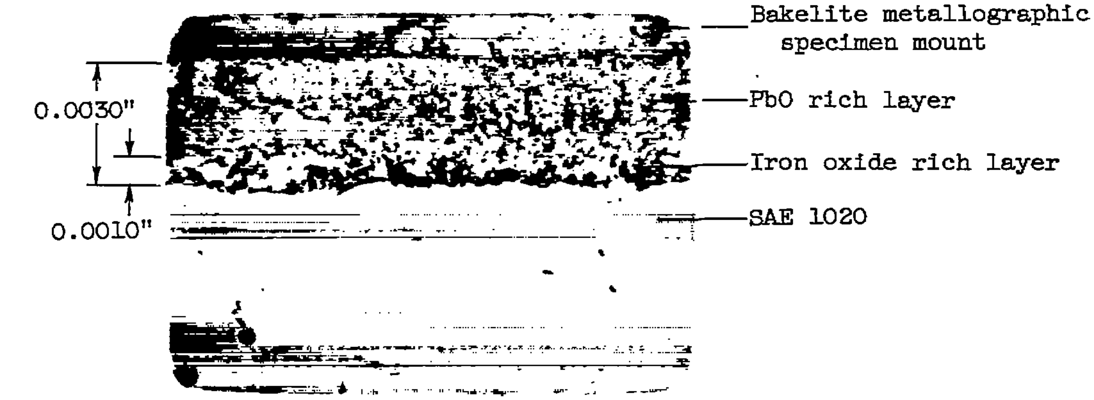
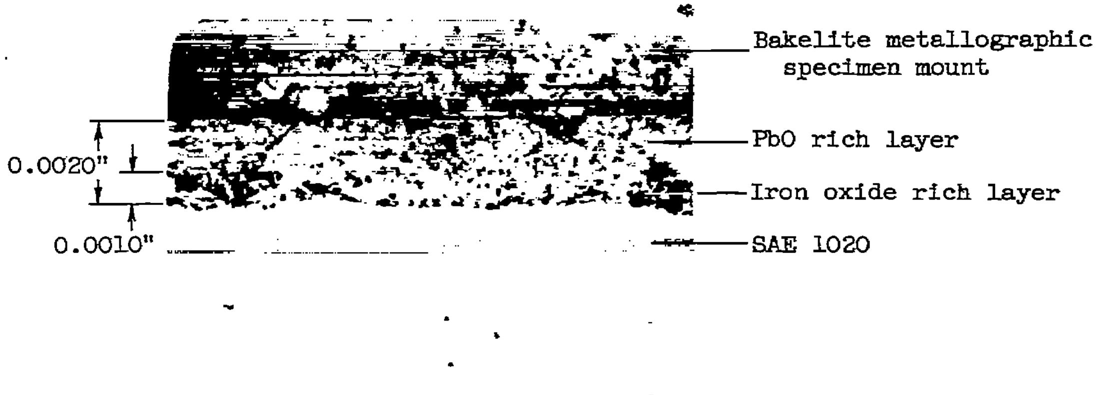


Figure 2. - Phase equilibrium diagram for $PbO-SiO_2$ system (ref. 5).



(a) Cross section through coating and base metal; unetched; X250; before test.

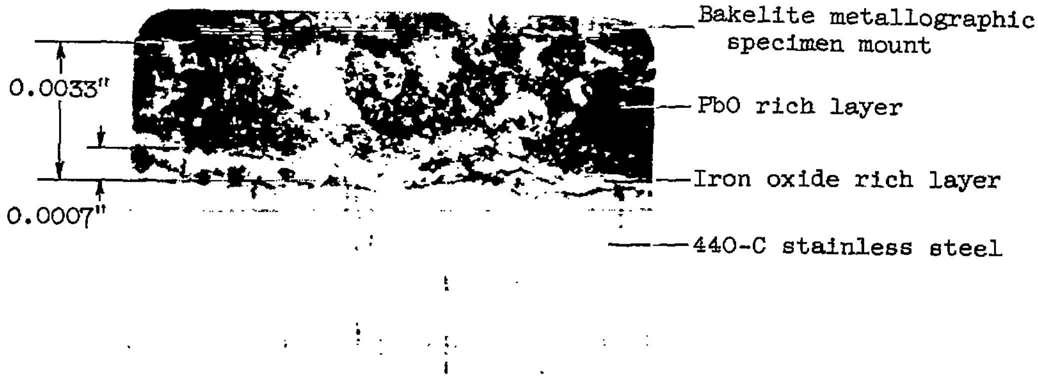


(b) Cross section through wear track and base metal; unetched; X250; after test.

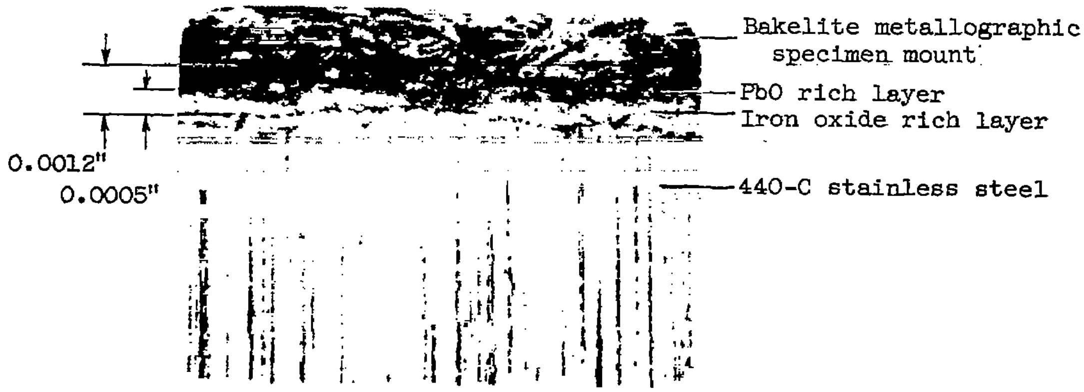
(c) Ground surface of coating and glazed wear track; unetched; X6$\frac{1}{2}$. C-44224

Figure 3. - Bonded PbO coating on SAE 1020 stainless steel.

4404



(a) Cross section through coating and base metal; unetched; X250; before test.



(b) Cross section through wear track and base metal; unetched; X250; after test.

C-44225

Figure 4. - Bonded PbO coating on 440-C stainless steel.

4404

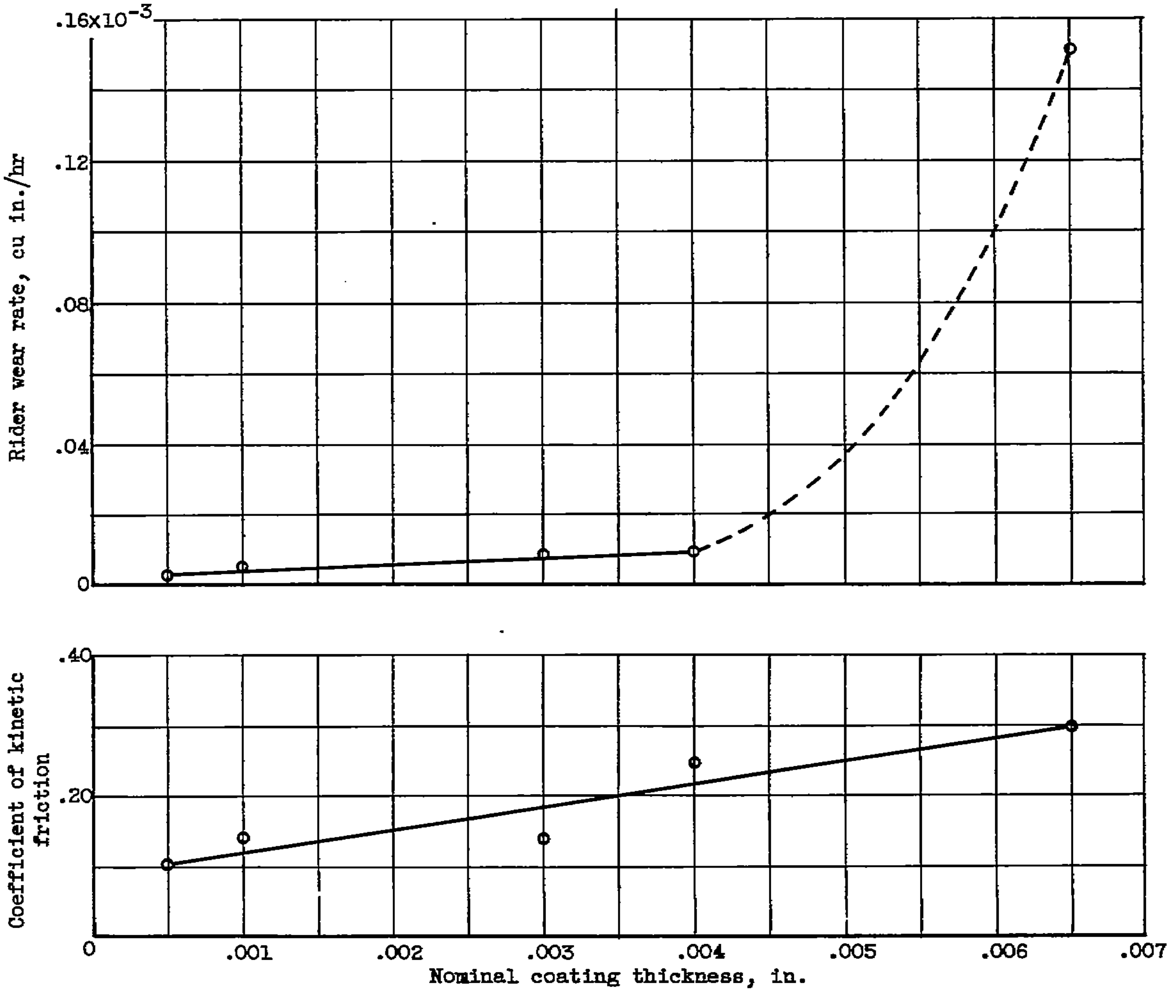
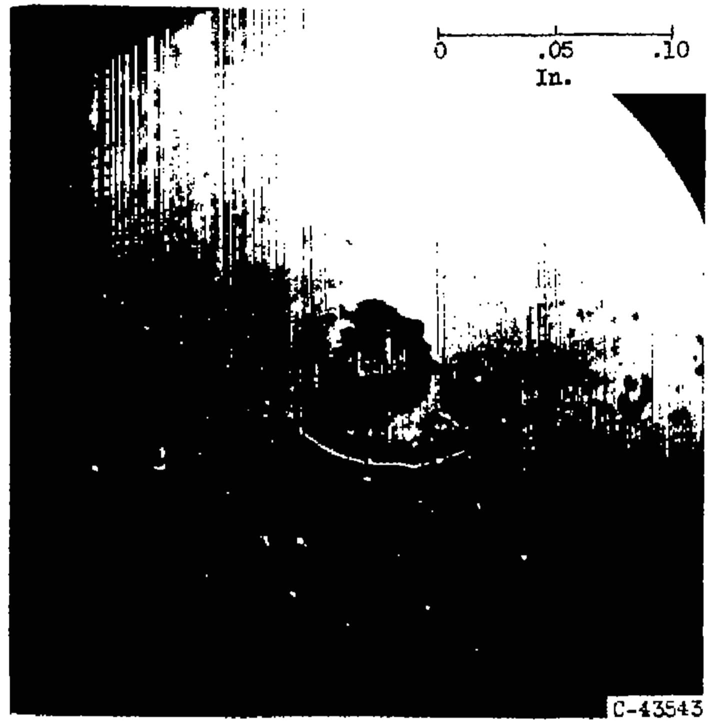


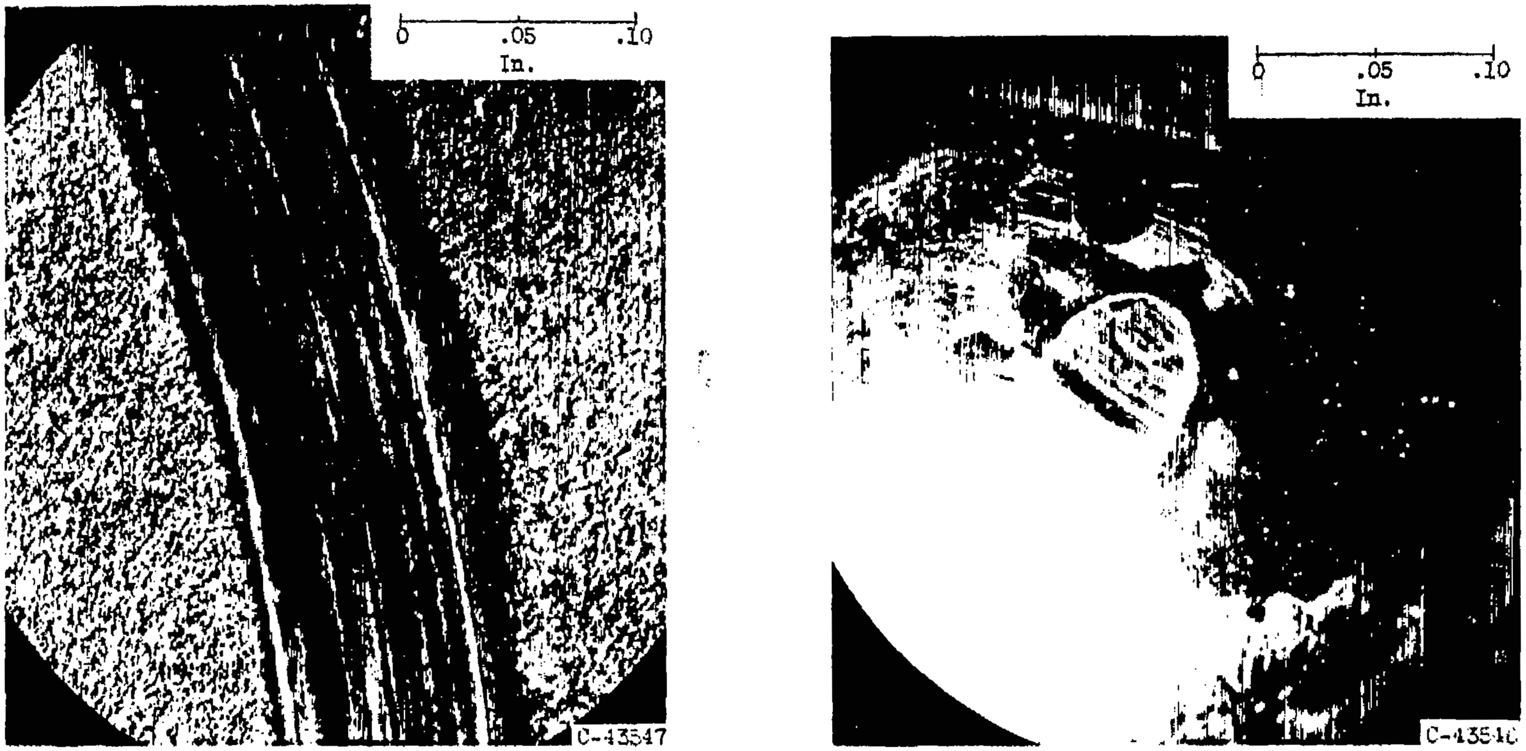
Figure 5. - Effect of coating thickness on friction and wear of cast Inconel sliding against bonded PbO in air at a temperature of 1250° F. Sliding velocity, 430 feet per minute; load, 1 kilogram; rider radius, 3/16 inch. Coatings prepared by fusion of 5 percent SiO_2 and 95 percent PbO on 440-C base metal.

(a) Glazed wear track formed on 0.0005-inch-thick coating, X15.



(b) Wear area formed on 3/16-inch-radius cast Inconel hemispherical rider during run against track shown in (a); X15.

Figure 6. - Wear surfaces produced by sliding cast Inconel against bonded PbO; base metal, 440-C stainless steel; frit composition, 95 percent PbO, 5 percent SiO_2; temperature, 1250° F; load, 1 kilogram; sliding velocity, 430 feet per minute; wear track diameter, 2 inches; test duration, 1 hour.

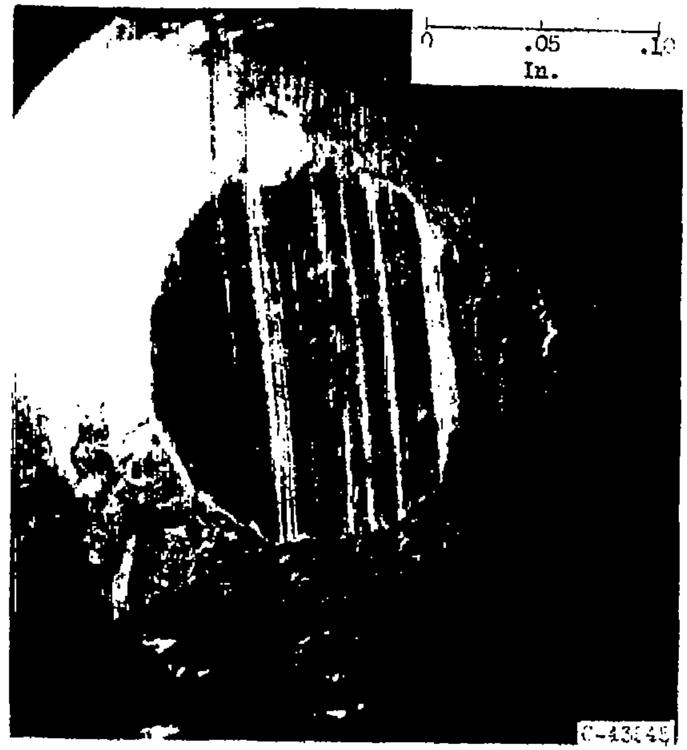


(c) Glazed wear track formed on 0.0010-inch-thick coating; X15.

(d) Wear area produced on 3/16-inch-radius cast Inconel hemispherical rider during run against track shown in (c); X15.

Figure 6. - Continued. Wear surfaces produced by sliding cast Inconel against bonded PbO; base metal, 440-C stainless steel; frit composition, 95 percent PbO, 5 percent SiO_2; temperature, 1250° F; load, 1 kilogram; sliding velocity, 430 feet per minute; wear track diameter, 2 inches; test duration, 1 hour.

(e) Partially failed, glazed wear track formed on 0.0065-inch-thick coating; X15.



(f) Wear area formed on 3/16-inch-radius cast Inconel hemispherical real rider during run against track shown in (e); X15.

Figure 6. - Concluded. Wear surfaces produced by sliding cast Inconel against bonded PbO; base metal, 440-C stainless steel; frit composition, 95 percent PbO, 5 percent SiO_2; temperature, 1250° F; load, 1 kilogram; sliding velocity, 430 feet per minute; wear track diameter, 2 inches; test duration, 1 hour.

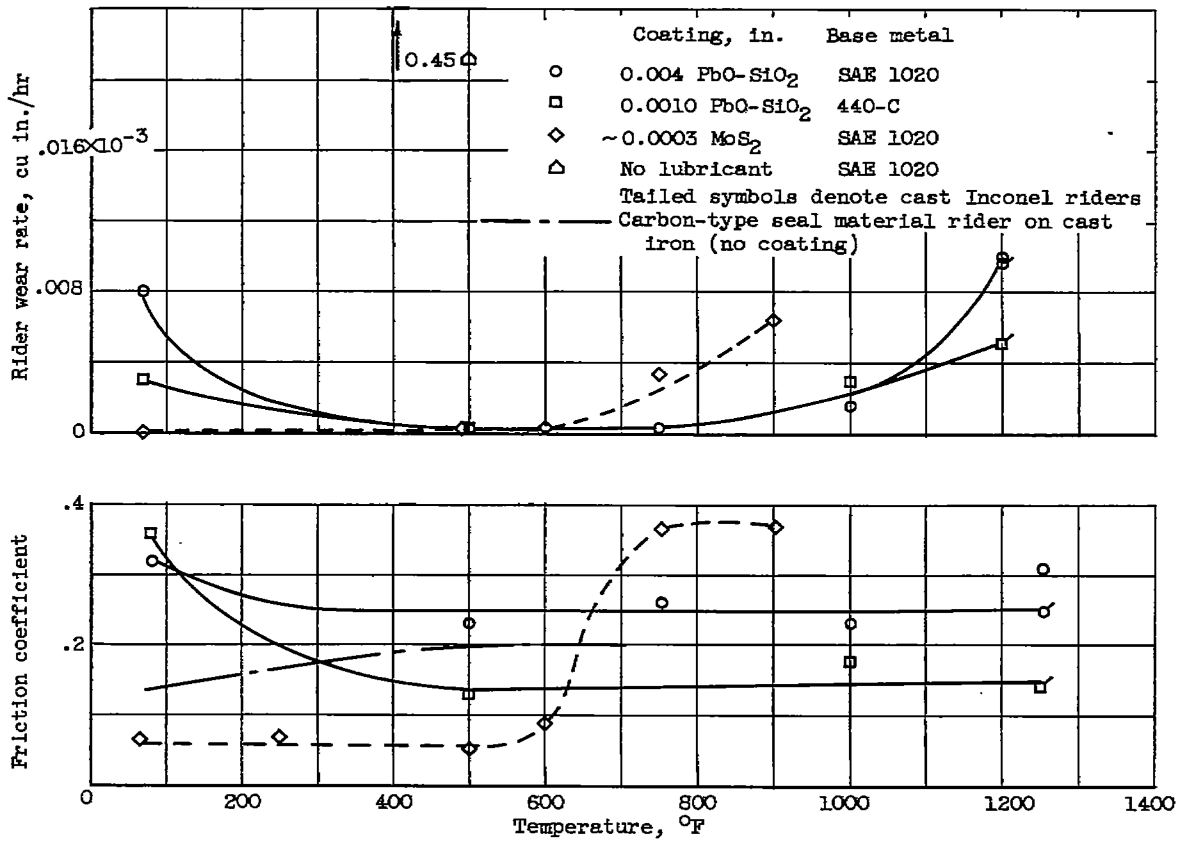


Figure 7. - Wear and friction at various temperatures with bonded solid-lubricant films. PbO coatings formed from mixtures of 95 percent by weight PbO and 5 percent by weight SiO_2. Sliding velocity, 430 feet per minute; load, 1 kilogram; radius of rider specimen, 3/16 inch. Rider specimens, hardened M-1 steel unless noted.

[redacted]

[redacted]